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PROGRESS REPORT

FOR

MARCH, 1951

BY

J. T. PINKSTON AND K. S. WILLSON

APRIL 6, 1951

THE HARSHAW CHEMICAL COMPANY

CLEVELAND, OHIO

CONTRACT NO. W-7405 ENG-276

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J. T. PINKSTON AND K. S. WILLSON

(Work done by D.A.Hammond, M.F.Rejdak, D.W.Barta and
K.S.Willson)

ABSTRACT

Extraction tests were carried out on a slurry made by digesting with nitric acid a mixture of black oxides and soda salt which was spiked with rare earths, molybdenum, boron, and iron. Those analytical data thus far available show that very minor amounts of all these impurities are dissolved in the TBP-Kerosene solvent when it is brought from the extractor substantially saturated with uranium, and that rare earth impurity can be further reduced by scrubbing.

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I. INTRODUCTION

The Brown Oxide Production Unit of the Atomic Energy Commission located in Plant "C" of the Harshaw Chemical Company has become increasingly inadequate because of the necessity of processing uranium feeds (black oxide and soda salts) of decreasing quality. The demand for increased output of brown oxide suitable for metal production from the above feeds, and ultimately from various rare earth containing feeds and Mg X materials, has necessitated a study of new methods of production.

This is the fourth progress report covering tests designed to show what changes are necessary to permit handling feeds of the types cited with production of a satisfactory product.

This report gives further results of the study of TBP-Kerosene extraction of uranium from slurries with particular attention to the purity of the extract from slurries heavily spiked with rare earths and other contaminants.

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II SURVEY OF PROBLEM

In previous progress reports (NYO-1452-1454), laboratory and semi-pilot plant tests were described which indicated that uranyl nitrate of high purity could be extracted from certain uranium containing slurries. Operating conditions were outlined for the extraction step whereby an essentially saturated solvent could be secured at the one end of the extraction process and a raffinate containing 0.01 g.U/l. or less at the other end. Data on scrubbing and stripping operations were given.

With the recent additional requirement that rare-earth containing feeds be handled to give a product suitable for the production of metal, new factors became important. This month, a study has been made of the operating conditions necessary to handle rare earth containing feeds, and to further study the effects of other contaminants in the process.

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III EXPERIMENTAL WORK

As reported previously, laboratory and semi-pilot plant tests had indicated a pure UNH could be secured by direct extraction of certain slurries. With the introduction of the necessity of handling rare earth materials, further tests have been carried out to determine what amount of rare earths and other contaminants occur in the loaded solvent and what scrubbing techniques may be necessary.

For the tests, a slurry was prepared from 30% Canadian Black Oxide, 30% Colorado Black Oxide and 40% Colorado Soda Salts and contained 314 g.U/l. Molybdenum and boron were added to the extent of 0.1% and iron to the extent of 1% based on the weight of uranium materials above. (Approximately 0.1% vanadium was also present in the feed itself). A rare earth oxide mixture was added to the extent of 2500 ppm. All of these "spike" materials were added in solution to the slurry and the slurry digested for 1 hour at 90°C.

A 15% TBP-Kerosene solvent was saturated by contacting it with 2 portions of slurry at 5:1 organic/solvent ratio. This saturated solvent was then scrubbed counter currently using various scrubbing solutions, varying number of stages, and varying solvent/aqueous ratios. Tests were carried out in separatory funnels and each test was run until equilibrium with respect to uranium content had been secured in all stages, before samples were secured.

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Samples of the solvent were analyzed in some instances. In two cases, the scrubbed solvent was stripped and the OK liquor boiled down to UNH for shot gun tests.

Some further limited tests have been carried out to assist the Engineering Department in designing the plant layout for the new process.

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IV RESULTS AND CONCLUSIONS

Analytical data on the work cited above are incomplete and further tests may be necessary to establish the best operating procedures. However, one shotgun test reported indicates a pure product (0.05 shotgun) can be obtained without any great difficulty. Partial results also indicate that the loaded solvent even before scrubbing will contain only very small amounts of rare earths. Many other contaminants present in the slurry will be low-either undetected or present only to the extent of a few ppm in the solvent. Boron of the order of 1.5 ppm, phosphorus of the order of 300 ppm (probably from the solvent) and silicon of the order of 30 ppm may be present although these elements may be derived in part from the glassware used in the tests. Scrubbing procedures thus far tested show little effect on these impurities although rare earth can be reduced to the order of ≤ 0.1 ppm for any individual reported member of the group.

Other data not yet available covering various types of scrubbing will be presented in subsequent reports.

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